Table I. Relative Nucleophilicities of 9-Substituted Fluorenyl Carbanions (C^-), 2-Naphthoxide Ions (O^-), and Thiophenoxide Ions (S^-) of the Same Basicity in Me_2SO Solution at 25 °C[a]

halide	k^{C^-}/k^{O^-}	k^{S^-}/k^{C^-}	k^{S^-}/k^{O^-}
$PhCH_2Cl$	25	550	13 500
BuCl	4	3000	12 000
BuI	55	1300	72 000

[a] Derived from extrapolations using Brønsted-type plots (e.g., Figure 1). Detailed rate and pK_a data for these reactions, together with reports of product studies, will be presented in the complete paper.

displacements from these Brønsted-type plots give the relative nucleophilicities of thiophenoxides, fluorenyl carbanions, and 2-naphthoxide ions, which are summarized in Table I.

Since the β_{Nu} values vary slightly and the relative rates are substrate dependent (Table I), it is impossible to define exact relative nucleophilicities. It is clear, nevertheless, that thianions are far more nucleophilic in Me_2SO than are carbanions or oxanions of the same basicity. Thiophenoxides are about 10^2–10^3 times more nucleophilic than fluorenyl carbanions and 10^4–10^5 times more nucleophilic than 2-naphthoxide ions, depending on the substrate.

The relatively high k^{S^-}/k^{O^-} ratio toward alkyl halides in hydroxylic solvents[1] can be ascribed in part to the much stronger solvation of O^- than S^- through hydrogen bonding. If the difference in pK_a values in Me_2SO vs. H_2O (ΔpK_a) is taken as a measure of the relative loss of hydrogen bonding solvation energies in changing from H_2O to Me_2SO, this amounts to about 4.4 kcal/mol for PhS^- (ΔpK = 3.2[6]) and 9.5 kcal/mol for 2-NpO^- (ΔpK_a = 6.9[6]). One would therefore expect 2-NpO^- (or PhO^-) ions to become more nucleophilic, relative to PhS^-, in changing the medium from H_2O (or MeOH) to Me_2SO, i.e., k^{S^-}/k^{O^-} should decrease. This does not seem to occur, however, since in MeOH $k^{PhS^-}/k^{PhO^-} \simeq 10^4$ toward MeI,[7,8] which is about the same order of magnitude that we observe in Me_2SO (Table I). Evidently, the decrease in k^{S^-}/k^{O^-} in Me_2SO caused by the absence of hydrogen bonding is counteracted by other factors that keep the ratio nearly constant in the two media.

The order of nucleophilicities, $F^- > Cl^-$, toward PrOTs in Me_2SO cited above appears at first sight to be contrary to the $O^- < S^-$ order. There is evidence to indicate, however, that F^- is a much stronger base in dipolar nonhydroxylic solvents than Cl^-.[9] The relative reactivities of Cl^- and Br^- toward PrOTs in Me_2SO, k^{Cl^-}/k^{Br^-} = 6.6,[4] correspond to that expected from their relative basicities.[10] Furthermore, fitting Parker's data for Cl^- and Br^- reacting with BuI in Me_2SO[2b] to our Brønsted line for the 9-CO_2Me-Fl^- family (β_{Nu} = 0.40) indicates that a Cl^- ion is about 10^4 times more nucleophilic than a carbanion of the same basicity. This suggests that the higher nucleophilicity of anions derived from second (and higher) row elements vs. first row elements of the same basicity is a general phenomenon in solution and is an intrinsic property.

(7) Pearson, R. G.; Sobel, H.; Songstad, J. *J. Am. Chem. Soc.* **1968**, *90*, 319–326.
(8) Since the basicity of PhO^- is 3.4 higher (in H_2O) than that of PhS^-, the k^{S^-}/k^{O^-} ratio will be about 10^5 for anions of equal basicity.
(9) Clark, J. H. *Chem. Rev.* **1980**, *80*, 429–452. Acidity measurements of HF in Me_2SO will be complicated by strong homohydrogen bonding between fluoride ion and hydrogen fluoride, i.e., $F^-\cdots H$–F.
(10) The acidities of HBr and HCl in Me_2SO are 0.92[11a] and 2.0,[11b] respectively.
(11) (a) McCallum, C.; Pethybridge, A. D. *Electrochim. Acta* **1975**, *20*, 815–818. (b) Benoit, R. L.; Buisson, C. *Electrochim. Acta* **1973**, *18*, 105–110.

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Registry No. 9-CO_2Me-Fl^-, 12565-94-5; 2-NpO^-, 15147-55-4; PhS^-, 13133-62-5; $MeC(CN)_2^-$, 78232-00-5; Ph_2CCN^-, 18802-83-0; 2,4,5-$Cl_3C_6H_2S^-$, 78232-01-6; 9-CO_2Me-2,7-Br_2-Fl^-, 73838-70-7; 3-$CF_3C_6H_4S^-$, 78232-02-7; 9-CN-Fl^-, 40052-38-8; 6-Br-2-NpO^-, 78232-03-8; 4,6-Br_2-2-NpO^-, 78232-04-9; 2-Br-9-CO_2Me-Fl^-, 73838-71-8; $PhCH_2Cl$, 100-44-7; BuCl, 109-69-3; BuI, 542-69-8; 2-naphthol, 135-19-3; 9-(carbomethoxy)fluorene, 3002-30-0.

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Tetraalkylammonium Trihydridocyanoborates. Versatile, Selective Reagents for Reductive Aminations in Nonpolar Media

Summary: Tetrabutylammonium cyanoborohydride or the combination of sodium cyanoborohydride with Aliquat 336 provides useful, convenient reagents for reductive amination of aldehydes and ketones in aprotic or protic media.

Sir: Trihydridocyanoborate (cyanoborohydride)[1] is well established as a mild, selective, acid-stable reducing agent for a variety of conversions including aldehydes and ketones to alcohols,[2] tosylhydrazones,[3] polar alkenes,[4] and alkyl halides[5] to hydrocarbons, and numerous carbon–nitrogen π-bond derivatives (imines, oximes, enamines) to amines.[2] This latter transformation has been particularly exploited as an excellent procedure for the reductive amination of aldehydes and ketones.[1,2,6] However, the commercially available sodium derivative suffers the limitation that solubility is restricted to a few polar protic (H_2O, low molecular weight alcohols), aprotic (Me_2SO, HMPA), or ether (THF, diglyme) solvents.[8] The reagent is almost totally insoluble and unreactive in most other useful solvents including CH_2Cl_2, $CHCl_3$, aromatic and aliphatic

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(2) Borch, R. F.; Bernstein, M. D.; Durst, H. D. *J. Am. Chem. Soc.* **1971**, *93*, 2897. Recently, the intermediacy of iminium ions in certain reductive aminations has been questioned: Tadanier, J.; Hallas, R.; Martin, J. R.; Stanaszek, R. S. *Tetrahedron* **1981**, *37*, 1309; Kapnang, H.; Charles, G.; Sondengam, B. L.; Hemo, J. H. *Tetrahedron Lett.* **1977**, 3469.
(3) Hutchins, R. O.; Maryanoff, B. E.; Milewski, C. A. *J. Am. Chem. Soc.* **1975**, *40*, 923.
(4) Hutchins, R. O.; Rotstein, D.; Natale, N. R.; Fanelli, J.; Dimmel, D. *J. Org. Chem.* **1976**, *41*, 3328.
(5) Hutchins, R. O.; Kandasamy, D.; Maryanoff, C. A.; Masilamani, D.; Maryanoff, B. E. *J. Org. Chem.* **1977**, *42*, 82.
(6) Other reagent systems recently introduced for reductive aminations include: (a) potassium hydridotetracarbonylferrate, Bodrini, G. P.; Panunzio, M.; Umani-Ronchi, A. *Synthesis* **1974**, *261*; (b) $NaBH_4/H_2SO_4$, Giumanini, A. G.; Chiavari, G.; Musiani, M. M.; Rossi, P. *Synthesis* **1980**, *743*; (c) the Leukart reaction; see, for example, Baeh, R. D. *J. Org. Chem.* **1968**, *33*, 1647; (d) $NaBH_4$ in carboxylic solvents, Schellenberg, K. A. *J. Org. Chem.* **1963**, *28*, 3259; Gribble, G. W.; Lord, P. D.; Skotnicki, J.; Dietz, S. E.; Eaton, J. T.; Jonson, J. L. *J. Am. Chem. Soc.* **1974**, *96*, 7812; Marchini, P.; Liso, G.; Reho, A.; Liboratore, F.; Moracci, F. M., *J. Org. Chem.* **1975**, *40*, 3453; (e) ion-exchange resin supported BH_3CN^-, Hutchins, R. O.; Natale, N. R.; Taffer, I. M. *J. Chem. Soc., Chem. Commun.* **1978**, 1088.
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(8) Wade, R. C.; Sullivan, E. A.; Berschied, J. R.; Purcell, K. F. *Inorg. Chem.* **1970**, *9*, 2146.

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Table I. Reductive Aminations with Tetraalkylammonium Trihydridocyanoborates

carbonyl	amine	hydride	solvent (time, h)	product (picrate mp, °C)[a]	% yield[b]
C_6H_5CHO	pyrrolidine	$NaBH_3CN$	CH_3OH (72)	*N*-benzylpyrrolidine (123–24)	70
		TBACB	CH_2Cl_2 (2.5)		76
		TBACB	THF (2.5)		75
		TBACB	hexane (2.5)		66
		TBACB	C_6H_6 (2.5)		64
		TBACB	CH_3CN (2.5)		58
		$NaBH_3CN$ Aliquat 336	CH_2Cl_2 (48)		84
	morpholine	$NaBH_3CN$ Aliquat 336	CH_2Cl_2 (48)	*N*-benzylmorpholine (185–86)	41
	$(CH_3CH_2)_2NH$[c] + $(CH_3CH_2)_2NH_2^+Cl^-$	$NaBH_3CN$ Aliquat 336	CH_2Cl_2 (42)	$C_6H_5CH_2N(CH_2CH_3)_2$ (116–17)	53
	$CH_3CH_2CH_2NH_2$	TBACB	CH_2Cl_2 (2)	$C_6H_5CH_2NHCH_2CH_2CH_3$	79
		$NaBH_3CN$ Aliquat 336	CH_2Cl_2 (48)		60
	$(CH_3)_2CHNH_2$	TBACB	CH_2Cl_2 (2.5)	$C_6H_5CH_2NHCH(CH_3)_2$	58
p-BrC_6H_4CHO	pyrrolidine	TBACB	CH_2Cl_2 (23)	*N*-(*p*-bromobenzyl)pyrrolidine (140–41)	83
m-ClC_6H_4CHO		TBACB	CH_2Cl_2 (21)	*N*-(*m*-chlorobenzyl)pyrrolidine (153–54)	89
2,6-$Cl_2C_6H_3CHO$		TBACB	CH_2Cl_2 (2.5)	*N*-(2,6-dichlorobenzyl)pyrrolidine (180–81)	57
		$NaBH_3CN$ Aliquat 336	CH_2Cl_2 (48)		79
p-NCC_6H_4CHO	pyrrolidine	TBACB	CH_2Cl_2 (17)	*N*-(*p*-cyanobenzyl)pyrrolidine (166–67)	58
$CH_3(CH_2)_8CHO$		$NaBH_3CN$	CH_3OH (46)	*N*-decylpyrrolidine (73–74)	67
		TBACB	CH_2Cl_2 (2.5)		64
	$(CH_3)_2CHNH_2$	TBACB	CH_2Cl_2 (42)	$CH_3(CH_2)_8CH_2NHCH(CH_3)_2$	52
$C_6H_5COCH_3$	pyrrolidine	$NaBH_3CN$	CH_3OH (70)	*N*-(α-methylbenzyl)pyrrolidine (125–26)	82
		TBACB	CH_2Cl_2 (72)		89
		TBACB	hexane (72)		69
		TBACB	C_6H_6 (72)		61
		$NaBH_3CN$ Aliquat 336	CH_2Cl_2 (48)		74
		$NaBH_3CN$ Aliquat 336	C_6H_6 (48)		70
	$CH_3NH_3^+Cl^-$	TBACB	CH_3OH (90)	$C_6H_5CH(CH_3)NHCH_3$ (182–83)	71
		TBACB	CH_2Cl_2 (90)		<5
	NH_4OAc	TBACB	CH_3OH (45)	$C_6H_5CH(CH_3)NH_2$	49
	$CH_3CH_2CH_2NH_2$	$NaBH_3CN$ Aliquat 336	CH_2Cl_2 (48)	$C_6H_5CH(CH_3)NHCH_2CH_2CH_3$	36
p-BrC_6H_4CHO	pyrrolidine	TBACB	CH_2Cl_2 (48)	*N*-(*p*-bromo-α-methylbenzyl)pyrrolidine (164–65)	82
cyclohexanone	pyrrolidine	$NaBH_3CN$	CH_3OH (72)	*N*-cyclohexylpyrrolidine (163–64)	71
		TBACB	CH_2Cl_2 (47)		90
cyclohexanone	pyrrolidine	$NaBH_3CN$ Aliquat 336	CH_2Cl_2 (48)	*N*-cyclohexylpyrrolidine	94
		$NaBH_3CN$ Adogen 464	CH_2Cl_2 (48)		78
		$NaBH_3CN$ Aliquat 336	$(C_2H_5)_2O$ (42)		73

	$NaBH_3CN$	hexane (42)		67
	Aliquat 336			
morpholine	TBACB	CH_2Cl_2 (2.5)	*N*-cyclohexylmorpholine (169–70)	58
	$NaBH_3CN$	CH_2Cl_2 (48)		52
	Aliquat 336			
$CH_3CH_2CH_2NH_2$	TBACB	CH_2Cl_2 (2.5)	cyclohexylpropylamine	55
$(CH_3)_2CHNH_2$	$NaBH_3CN$	CH_2Cl_2 (48)	cyclohexylisopropylamine	48
	Aliquat 336			
$(C_2H_5)_2NH^c$ +	$NaBH_3CN$	CH_2Cl_2 (48)	*N*,*N*-diethylcyclohexylamine	36
$(C_2H_5)_2NH_2^+Cl^-$	Aliquat 336			
4-*tert*-butylcyclohexanone	TBACB	CH_3OH (45)	4-*tert*-butylcyclohexanol (~145, mixture)	73 (67t, 33c)
$CH_3(CH_2)_5COCH_3$	$NaBH_3CN$	CH_2Cl_2	2-pyrrolidinyloctane (86–87)	73
	Aliquat 336			
$C_6H_5NH_2$	TBACB	CH_2Cl_2	$CH_3(CH_2)_5CH(CH_3)NHC_6H_5$	31
	$NaBH_3CN$	CH_3CN (48)	$C_6H_5N(CH_3)_2$	66
	$NaBH_3CN$	CH_2Cl_2		61
	Aliquat 336			

[a] All known products were identified by comparisons with authentic samples. New compounds showed IR, NMR, and elemental analysis consistent with the assigned structures. [b] Isolated and purified by distillation. [c] No added acid.

hydrocarbons, and diethyl ether.

To circumvent the solubility problem and hence augment the utility of cyanoborohydride, we have explored the use of the tetrabutylammonium derivative[9] and other phase-transfer techniques[10] for typical cyanoborohydride reductions in nonpolar media.[5,9,11] This communication reports the successful application of phase transfer to reductive amination, which extends the useful media for these conversions to include most common organic solvents, including CH_2Cl_2, hexane, benzene, and diethyl ether.

Tetrabutylammonium cyanoborohydride (TBACB), prepared as previously described,[9,11] is an air- and moisture-stable crystalline solid (mp 144–45 °C) which, in contrast to the sodium counterpart, is not hygroscopic. Phase transfer was also used to solubilize $NaBH_3CN$ by employing Aliquat 336, an inexpensive liquid reagent composed of methyltrialkylammonium chlorides with C_8–C_{10} chains. Successful reductive aminations were obtained under a variety of conditions, but the most convenient consisted of simply dissolving the aldehyde or ketone (10 mmol), amine (60 mmol), and TBACB (7 mmol) or $NaBH_3CN$ (7 mmol) plus Aliquat 336 (7 mmol) in 21 mL of solvent followed by addition of HCl (20 mmol), conveniently added as a 2.5–5.0 N solution in methanol or other solvent. Approximately 1 g of 4A molecular sieves was added (to absorb H_2O formed), and the mixture was stirred at ambient temperature. Progress of the reactions could be followed by monitoring the disappearance of the carbonyl by IR. Upon completion, isolations were accomplished in standard fashion (experimental), the products purified by short-path distillation, and identified by comparison (IR and/or NMR) with authentic samples.

The results for a range of carbonyls and amines are presented in Table I. Examples using the standard method ($NaBH_3CN$, CH_3OH, 2–3 days)[2] are included for comparisons. As illustrated, aromatic and aliphatic aldehydes and ketones react readily with unhindered primary and secondary amines to afford respectable to excellent isolated yields of amines in reasonable times, usually 2.5–24 h for aldehydes and 24–48 h for ketones. Two limitations were encountered. Relatively hindered secondary amines (i.e., diethylamine) reacted only reluctantly with ketones and gave inferior yields (<40%) of amine products. Also ammonium salts ($NH_4^+X^-$, $RNH_3^+X^-$) generally failed to react in aprotic solvents in which solubility is a problem. In such cases, methanol solvent is clearly superior.[2]

The general reaction procedure is illustrated for the preparation of *N*-cyclohexylpyrrolidine. To a solution containing pyrrolidine (4.26 g, 60 mmol) in 21 mL of CH_2Cl_2 was added HCl (20 mmol, 8 mL of a 2.5 N solution in CH_3OH) followed by cyclohexanone (0.98 g, 10 mmol), $NaBH_3CN$ (0.44 g, 7 mmol), and Aliquat 336 (2.93 g, 7 mmol). Approximately 1 g of 4A molecular sieves was added, and the mixture was stirred at room temperature for 48 h. The mixture was filtered, the filtrate acidified (methyl orange indicator), and the solvent removed on a rotary evaporator. The residue was taken up with 10 mL of H_2O and extracted with three 20-mL portions of ether

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(10) For excellent, general reviews of phase-transfer reactions, including reductions, see Weber, W. P.; Gokel, G. W. "Phase Transfer Catalysis in Organic Synthesis"; Springer-Verlag: New York, 1977; Keller, W. E. "Compendium of Phase-Transfer Reactions and Related Synthetic Methods"; Fluka AG, Ch-9470 Buchs, Switzerland, 1979.

(11) Hutchins, R. O.; Kandasamy, D. *J. Org. Chem.* **1975**, *40*, 2530.

(discarded). The aqueous phase was basified (solid KOH, phenolphthalein indicator), 20 mL of brine was added, and the mixture was extracted exhaustively with ether. These combined extracts were dried ($MgSO_4$), concentrated, and distilled in a Kugelrohr apparatus to yield 1.43 g (94%) of *N*-cyclohexylpyrrolidine, identified by comparison (IR) with an authentic sample. GLC analysis indicated >98% purity.

In conclusion, phase-transfer techniques greatly augment the utility of cyanoborohydride for reductive aminations of carbonyls and complement analogous conversions in protic media.

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Registry No. Cyclohexanone, 108-94-1; 4-*tert*-butylcyclohexanone, 98-53-3; pyrrolidine, 123-75-1; morpholine, 110-91-8; *N*-benzylpyrrolidine picrate, 78064-90-1; *N*-benzylmorpholine picrate, 58531-53-6; *N*-(*p*-bromobenzyl)pyrrolidine picrate, 78064-91-2; *N*-(*m*-chlorobenzyl)pyrrolidine picrate, 78064-93-4; *N*-(2,6-dichlorobenzyl)pyrrolidine picrate, 78064-95-6; *N*-(*p*-cyanobenzyl)pyrrolidine picrate, 78064-97-8; *N*-decylpyrrolidine picrate, 78064-98-9; *N*-(α-methylbenzyl)pyrrolidine picrate, 78064-99-0; *N*-(*p*-bromo-α-methylbenzyl)pyrrolidine picrate, 78065-00-6; *N*-cyclohexylpyrrolidine picrate, 33109-41-0; *N*-cyclohexylmorpholine picrate, 33109-39-6; cyclohexylpropylamine picrate, 78065-01-7; cyclohexylisopropylamine picrate, 2499-05-0; *N,N*-diethylcyclohexylamine picrate, 78065-02-8; *N*-(4-*tert*-butylcyclohexyl)pyrrolidine picrate, 78065-03-9; 2-pyrrolidinyloctane picrate, 42367-34-0; TBACB, 43064-96-6; $NaBH_3CN$, 25895-60-7; C_6H_5CHO, 100-52-7; *p*-BrC_6H_4CHO, 1122-91-4; *m*-ClC_6H_4CHO, 104-88-1; 2,6-$Cl_2C_6H_3CHO$, 83-38-5; *p*-NCC_6H_4CHO, 105-07-7; $CH_3(CH_2)_8CHO$, 112-31-2; $C_6H_5COCH_3$, 98-86-2; $CH_3(CH_2)_5COCH_3$, 111-13-7; CH_2O, 50-00-0; $(CH_3CH_2)_2NH$, 109-89-7; $(CH_3CH_2)_2NH\cdot HCl$, 660-68-4; $CH_3CH_2CH_2NH_2$, 107-10-8; $(CH_3)_2CHNH_2$, 75-31-0; $CH_3NH_2\cdot HCl$, 593-51-1; NH_4OAc, 631-61-8; $C_6H_5NH_2$, 62-53-3; $C_6H_5CH_2NHCH_2CH_2CH_3$ Picrate, 78065-04-0; $C_6H_5CH_2NHCH(CH_3)_2$ Picrate, 68723-39-7; $CH_3(CH_2)_8CH_2NHCH(CH_3)_2$ Picrate, 78065-06-2; $C_6H_5CH(CH_3)NHCH_3$ Picrate, 78065-07-3; $C_6H_5CH(CH_3)NH_2$ Picrate, 78065-08-4; $C_6H_5CH(CH_3)NHCH_2CH_2CH_3$ Picrate, 78065-09-5; $CH_3(CH_2)_5CH(CH_3)NHC_6H_5$ Picrate, 78065-10-8; $C_6H_5N(CH_3)_2$ Picrate, 7510-42-1; $C_6H_5CH_2N(CH_2CH_3)_2$ Picrate, 78065-11-9.

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Preparation of Hydroxy Crown Ethers by Reactions of Diphenols with Epichlorohydrin

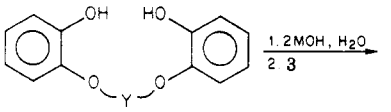
Summary: Reactions of epichlorohydrin with appropriate diphenols in basic aqueous media produce good yields of crown ethers with hydroxyl groups attached to the crown ether ring.

Sir: Synthetic routes to crown ethers which bear pendant functionality have received considerable attention.[1] The functional groups may provide additional liganding atoms for cation complexation,[2,3] serve as sites for further structural elaboration,[3-7] or function as attachment points for binding crown ethers to polymers.[5,6,8] For such purposes, alcohol groups are often the most versatile. Several methods for the preparation of crown ethers with one or more pendant alcohol-containing groups have appeared.[1,2,4,6,8,9] However, many are rather complicated multistep syntheses. We now report the facile ring closure of diphenols **1a-e** to hydroxy crown ethers **2a-e**, using epichlorohydrin (**3**) in basic aqueous media (eq 1).

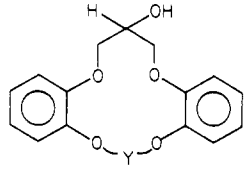
Table I. Formation of Hydroxy Crown Ethers by Reactions of Diphenols with Epichlorohydrin[a]

diphenol	M of MOH	hydroxy crown ether[b] identity	yield, %	mp, °C
1a	Na	2a	60	122-123
1b	K	2b	39	73-74
1c	Li	2c	50	142-143
1d	Li	2d	51	153-154

[a] In a typical procedure, 15 mM of **1**, 30 mM of MOH, and 350 mL of water are stirred under nitrogen at 90-95 °C until solution is achieved. After the solution is cooled to 50 °C, 15 mM of **3** is added over a period of 3 h. Upon completion of the addition, the reaction mixture is stirred at 50 °C for an additional 3-5 h and then cooled to room temperature. The precipitate (for **1b** the oil solidifies when the reaction mixture is cooled to 0 °C) is filtered and dissolved in CH_2Cl_2. The CH_2Cl_2 solution is washed with water and dried over $MgSO_4$, and the solvent is evaporated in vacuo to give the crude **2**. The crude product is placed on top of a short silica gel column and eluted with Et_2O to separate **2** from a small amount of Et_2O-insoluble polymeric material. [b] Satisfactory elemental and spectral analyses were obtained for **2a-d**.



1a,[10-12] Y = $CH_2CH_2OCH_2CH_2$
b,[11,12] Y = $CH_2CH_2OCH_2CH_2OCH_2CH_2$
c,[13] Y = CH_2CH_2
d,[12] Y = $CH_2CH_2CH_2$
e,[9] Y = $CH_2CH(OH)CH_2$
f, Y = $CH_2C(O)NHCH_2CH_2NHC(O)CH_2$



(1)

2a-f

In an isolated report, Ashby et al.[14] noted that reaction of **3** and the amidic diphenol **1f** under basic conditions produced **2f** in very low yield. We were therefore surprised to discover that a slow addition of **3** to an aqueous solution of the disodium salt of **1a** produced a 60% yield of the

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